

CHAPTER 4.9

ABERRATION-CORRECTED
ELECTRON MICROSCOPY

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Microscopy allows us to observe objects we cannot see with our eyes alone. With a light microscope, we can distinguish objects at the scale of the wavelengths of visible light just under a micrometer. Around 1870 Ernst Abbe, who laid the foundation of modern optics, suggested that the resolution of a microscope would improve by using some yet-unknown radiation with shorter wavelengths than visible light, that is, below 390 nanometers ($1\text{ nm} = 10^{-9}\text{ m}$). Electrons can have wavelengths near 1 picometer ($1\text{ pm} = 10^{-12}\text{ m}$) and should therefore allow atoms to be distinguished, since they are typically at least a few hundred pm apart. In this oversimplified view, further decreasing the wavelength of the radiation should allow us to increase the resolution of an electron microscopy even more. However, this can only be done by increasing the energy of the radiation, which would ultimately destroy the samples. Other factors also affect the resolution of electron microscopes, among them non-ideal imaging properties of electromagnetic lenses, which result in false images. It took more than half a century to understand and control these aberrations and separate objects less than 1 Ångström ($1\text{ Å} = 10^{-10}\text{ m}$) apart.

At the Technische Universität Berlin in 1931, Max Knoll and his doctoral student Ernst Ruska showed that magnetic coils could be used as lenses for electrons and thereby cleared the path to developing

transmission electron microscopy (TEM).¹⁹ In TEM, a broad electron beam impinges on a sample and the electrons exiting the sample are magnified using an electro-optical lens. These “transmitted” electrons carry information about the structure of the material in the sample. A year after their first publication, the resolution limit of the light microscope was surpassed. The wavelength of electrons can be controlled by their accelerating voltage. It is about 4 pm for a 100 keV electron, smaller than an atomic diameter. In comparison, green light has a wavelength of about 550 nm and good light microscopes can separate two objects about 300 nm apart, which corresponds to about 1,000 atomic diameters.

Subsequently, Max Knoll and Manfred von Ardenne, who headed his private research laboratory, the Forschungslaboratorium für Elektronenphysik in Berlin-Lichterfelde made early attempts to scan a specimen with a fine electron beam and thereby also create an image.^{18,33} Although conventional TEM continued to dominate electron microscopy for over fifty years; the scanning technique was further developed in the 1960s by Albert Crewe and his collaborators in Chicago,⁹ leading to the imaging of heavy atoms in Scanning Transmission Electron Microscopy (STEM).⁸ This led to a revolution in sub-Ångström imaging in the late 1990s, when correcting aberrations associated with electro-optical lenses (as outlined in the next section) finally became possible. Today, STEM imaging is ubiquitous and routinely used in materials science and engineering as well as in condensed matter physics and chemistry. Many of the entries in this volume examine tools that were introduced to the materials research community, rapidly gained widespread acceptance, and then continued to slowly evolve. Other tools, however, struggled to live up to their promises, sometimes over many decades, before finally achieving success. STEM is a particularly clear instance of the latter category.

Theoretical Reasons for Doubt and Hope

In 1926 Hans Busch from Jena University described the focusing of electrons in a manner similar to how glass lenses can focus visible

1 light. No glass or electromagnetic lens is perfect, but in light optics
2 aberrations can be corrected by a series of convex and concave lenses.
3 In electron optics this is not possible because there are no concave
4 lenses. Thus, new ways of correcting for the aberrations caused by
5 round electron lenses had to be developed.

6 The resolution of a STEM depends on the size of the electron
7 beam one scans across the sample. In turn, the size of the beam
8 depends on three main electro-optical effects. The first is the diffrac-
9 tion limit, which relates the resolving power to distinguish two
10 objects and is proportional to the wavelength and inversely propor-
11 tional to the illumination angle. Maximizing resolution would thus
12 aim for high energies and small wavelengths of electrons as men-
13 tioned above and large illumination angles. The second is the spher-
14 ical aberration of lenses: in STEM imaging, the smallest achievable
15 electron probe is proportional to a measure of the deviation from
16 perfect lensing called the constant of spherical aberration and the
17 third power of the illumination angle. In a simple ray-tracing pic-
18 ture, this can be understood as creating a disk of least confusion
19 before the image plane (see Figure 4.9.1). One should attempt to
20 minimize both the wavelength and spherical aberration using appro-
21 priate optics. In contrast to the diffraction limit, one would aim
22 for the smallest possible illumination angle. Third, the chromatic
23 aberration in the image plane is due to the presence of electrons
24 with different energies coming from their source and creating a disk
25 of confusion whose size is proportional to the chromatic aberration
26 constant and the width of the energy distribution. At small illumi-
27 nation angles, the diffraction limit determines the resolution, while
28 at larger ones the spherical aberration dominates, provided that the
29 spherical and chromatic aberration constants are of similar orders
30 of magnitude and the energy spread of the electrons is about 1 eV.
31 STEM imaging using energies above 100 kV will have spherical and
32 not chromatic aberration as the main limiting effect.

33 Early work by Otto Scherzer at the Technische Hochschule Darm-
34 stadt pointed to the unavoidable fact that round electron lenses have
35 positive spherical and chromatic aberration effects, which will limit
36 the resolution of an electron microscope (“Scherzer limit”) to about

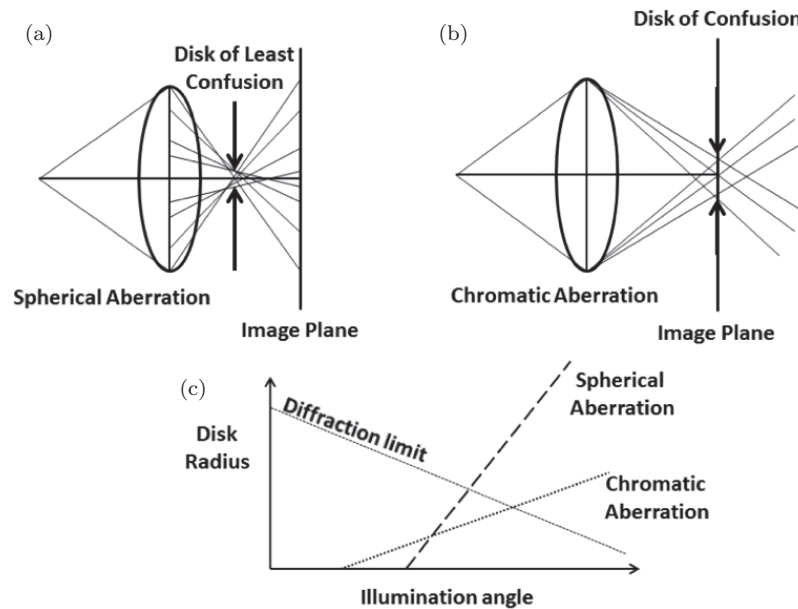




Fig. 4.9.1. Ray-tracing of spherical (a) and chromatic (b) aberration effect in cylindrical electro-optical lenses and their dependence on the illumination angle for acceleration voltages higher than 100 kV. An electrostatic hexapole sketched to the right of the ray tracing of the spherical aberration effect allows for its correction as it displaces the electrons with electric fields as indicated. Part (c) shows how diffraction limit, spherical aberration, and chromatic aberration vary with the radius of the disk of least confusion and the angle of the illuminating electron beam.

1 one hundred times the electron wavelength, which in a TEM is near
 2 2–2.5 picometers.³⁰ In electron optics, round lenses will always dis-
 3 play a positive aberration and no negative aberration is possible.
 4 But in a groundbreaking paper in 1947, Scherzer showed that both
 5 chromatic and spherical axial aberrations could be corrected by devi-
 6 ating from round lenses with a rotationally symmetric electromag-
 7 netic field using an electromagnetic multipole.²⁹ He predicted, based
 8 on a “gut-feeling” (German: *rein gefuehlsmaessig*) that this resulted
 9 in a negative correction effect to counteract the positive aberration.
 10 Think of a multipole as a geometrical figure approximating a sphere
 11 by placing points on the surface of the sphere and connecting them.
 12 Rotating a quadrupole, hexapole, or octupole, made of electrical

1 fields pointing in different directions, only approximates the rota-
2 tional symmetry of the sphere and it is this small deviation from the
3 sphere's shape that  needed for aberration correction. A sketch is
4 given in Figure 4.9.1.

5 **The First Arduous Steps toward Aberration** 6 **Correction**

7 Between 1949 and 1954, Robert Seeliger, a student of Scherzer,
8 was the first who attempted to build a corrector for a TEM com-
9 prised of two round lenses and three octupoles; however, he failed
10 to overcome mechanical and electromagnetic instabilities.³¹ Never-
11 theless, he proved Scherzer's conjecture that such a multipole cor-
12 rector leads to a negative spherical aberration needed to compensate
13 for the positive one of the round objective lens. Seeliger's correc-
14 tor was subsequently moved to the University of Tübingen where
15 Gottfried Möllenstedt intentionally enhanced the spherical aberr-
16 ation by using an unusually large illumination angle to degrade the
17 resolution (see Figure 4.9.1).²⁴ Using octupoles, he then improved
18 the resolution by about a factor of seven and significantly increased
19 the contrast. Largely ignored work by Möllenstedt's PhD student
20 Werner E. Meyer at the University of Tübingen showed that mis-
21 alignment, static imperfections, and charging, and alternating exter-
22 nal electromagnetic fields, as well as mechanical instabilities, were
23 the main roadblocks for aberration correction.²³ This proved to be
24 correct until the 1990s! Geoffrey Archard,¹ working at the Associ-
25 ated Electrical Industries (AEI) research laboratory at Aldermaston
26 Court, built on this early work establishing that cylindrical lenses can
27 be substituted by quadrupoles. He pointed out that an early attempt
28 by Jack C. Burfoot at the University of Cambridge could be simplified
29 by using a sequence of four optical elements: a quadrupole-octupole,
30 a round lens-octupole, a second quadrupole-octupole, followed by a
31 quadrupole. In 1964 Hans Deltrap built the first quadrupole-octupole
32 corrector to eliminate spherical aberration.^{5,12} However, the resolu-
33 tion was limited not by the spherical resolution, but other qualities
34 of the lenses at that time and his corrector was only tested on an
35 electron-optical bench not in an electron microscope.

1 An important international workshop took place in 1966 at
2 Argonne National Laboratory, at which the design of a high-voltage,
3 high-resolution electron microscope with a quadrupole-octupole cor-
4 rector was proposed but subsequently not funded. In 1971, Harald
5 Rose at TU Darmstadt, after showing that all correctors to date
6 suffered from off-axis coma, a “comet-like blur away from the opti-
7 cal axis,” developed a corrector that could simultaneously correct
8 for chromatic and spherical aberration.²⁷ In 1972 Crewe and Vernon
9 Beck made new attempts to correct for spherical aberration of a
10 STEM using a quadrupole-octupole corrector alleviating some of the
11 difficulties encountered in the Darmstadt project.⁷ However, they
12 were unable to eliminate aberrations due to inhomogeneity of the
13 metal used in the lens and the lack of fast diagnostic and control
14 electronics to establish suitable optical settings for the many param-
15 eters.  such electronics, no feedback control was possible. At the
16 International Congress on Electron Microscopy in Toronto in 1978,
17 Scherzer quipped that the resolution of the best uncorrected TEMs
18 “is clearly limited by the unavailability of the necessary funds.”²⁸ The
19 Darmstadt project continued until Scherzer’s death in 1982, when it
20 was terminated despite significant progress.⁴

21 Up to this point, techniques using sextupole correctors had been
22 ignored since Peter Hawkes had shown in 1965 that the large
23 second-order aberrations prevent the exploitation of the favorable
24 third-order aberrations to correct for spherical aberration of round
25 lenses.¹⁶ In an important breakthrough, Beck showed in 1979 that
26 one can arrange sextupoles in a way to suppress this large second-
27 order aberration.³ This design was subsequently improved by Rose
28 and later morphed into a prototype of the sextupole correctors
29 which are now produced commercially by the CEOS Company in
30 Heidelberg.²⁶

31 **The Darkest Hour . . . and a New Dawn**

32 In the late 1980s “aberration fatigue” set in after the Darm-
33 stadt project ended with Scherzer’s death. Crewe gave up, saying
34 “unfortunately, we could never make the corrector work. . . . After

1 many heartbreaking attempts, we were forced to admit defeat,” and
2 the US National Science Foundation decided to no longer fund efforts
3 to build correctors.¹⁵ The materials science community, which had
4 always been seen as the main user of high-resolution imaging, set
5 their hopes on high-voltage electron microscopy where resolution was
6 simply enhanced by reducing the wavelength of the electron beam
7 (see diffraction-limit above and Figure 4.9.1c) by going to accelera-
8 tion voltages of 1 million volts and higher. These gigantic instruments
9 reached Ångström resolution and the reduced electron inelastic scat-
10 tering at such high energies allowed the use of thicker samples. How-
11 ever, atom displacement damage (“knock-on” damage) destroyed the
12 samples, often within minutes.

13 There is a certain irony that funding agencies in Europe, Japan,
14 and the United States were prepared to invest an order of magni-
15 tude more in high-voltage instruments (one microscope would cost
16 tens of millions of US dollars) than was ever allocated for aberration
17 correction of electron lenses. During these dark days for the
18 aberration-correction community, it was realized that the required
19 machining, manufacturing, and positioning of the many required
20 optical elements was beyond what could reproducibly be achieved
21 at that time. Furthermore, neither the stable hardware (that is,
22 power supplies) nor the software existed to excite the various mul-
23 tipoles and other optical elements and integrate them with diag-
24 nostic tools based on fast detector readout to ensure feedback for
25 auto-tuning algorithms. As Harold Rose was quoted by Knut Urban,
26 “it took more time to adjust a specific electron optical state than
27 this would have time to last.”³² Also, a quadrupole-octupole cor-
28 rector is not very suitable for conventional TEM as one needs to
29 correct over the whole region imaged. A much more suitable imaging
30 system would be to correct a probe-forming SEM or STEM. For-
31 tunately, the electron optics community proved to be resilient and
32 was ultimately able to complete the revolution in high-resolution
33 electron imaging after a long march of over four decades. Three
34 main efforts in Europe funded internally by the European Molecular
35 Biology Laboratory (EMBL), privately by the Volkswagen Founda-
36 tion and by the Paul Instrument Fund of the Royal Society in the

1 United Kingdom provided the funds needed to realize imaging at the
2 atomic scale.

3 Due to the intrinsic difficulty in applying quadrupole correctors to
4 TEM, Max Haider convinced management that a SEM with spherical
5 and chromatic corrections aligned with the mission of EMBL as it had
6 the potential to be used for imaging larger biomolecules. EMBL had
7 a tradition of instrument development and could support this effort
8 by in-house funding. With Joachim Zach, Haider proved the work-
9 ing principle of a quadrupole-octupole corrector using a low-voltage
10 SEM. Despite its intrinsic lower resolution compared to a TEM,
11 the instrument's resolution was improved from 5.6 to 1.8 nm.^{34,35}
12 Their design was a corrector based on Hardy's thesis from 1967 in
13 Cambridge.

14 Parallel to these efforts Haider, Rose, and Urban lobbied for and
15 received high-risk funding from the Volkswagen Foundation which
16 allowed them to design, build, and test a hexapole-based corrector
17 at the EMBL in Heidelberg. Rose's corrector used a telescope round
18 lens doublet and two identical sextupoles, one centered at the front
19 focal point of the first round lens and the other on the back focal
20 point of the second round lens. The hexapole arrangement cancels
21 out all second order path deviations, whereas the third order rota-
22 tionally symmetric path deviations add up. The result is a nega-
23 tive spherical aberration proportional to the square of the hexapole
24 strength compensating the positive one of the objective lens. The
25 prolonged struggle to secure funding is described in detail in Urban's
26 2015 paper. The final decision by the Volkswagen foundation was
27 made by a one-vote majority and only part of the requested funding
28 was granted; the rest was contingent on achieving a critical milestone
29 showing that aberration correction could be achieved on a conven-
30 tional TEM, a worst-case test as mentioned above. First images were
31 obtained on a Philips CM200 microscope with a Rose corrector in
32 Heidelberg by Bernd Kabius on June 24, 1997, before the instrument
33 was shipped to Jülich since EMBL management had decided to shut
34 down in-house electron microscopy development. He showed that one
35 could resolve dumbbells (two atoms in close proximity in a projec-
36 tion) 1.4 Å apart when viewing along the (110) direction in a GaAs

sample. A paper was submitted first to *Nature* and then *Science* and initially rejected by both but finally accepted in *Nature*.¹⁴

During the same period, Ondrej Krivanek and Niklas Delby continued to work on adapting quadrupole-octupole correctors to a Vacuum Generator (VG) STEM at the Cavendish Laboratory in Cambridge. They were funded by the Royal Society (Paul Instrument Fund) and now had access to high speed diagnostic and control electronics to tune the corrector. Furthermore, they installed additional corrector coils to cancel parasitic aberrations. These improvements allowed them to present initial results in 1997 — one hundred years after the discovery of the electron by J. J. Thomson.²¹ Impressive results using dark field (DF) imaging were published in 1998.²⁰ Subsequently Krivanek and Delby moved to Seattle and started the company NION (Niklas and ONdrej). In 2000 their MARK-2 corrector was working and installed in a VG STEM at a specially shielded site at IBM where it allowed DF images at a resolution of 1.4 Å to be recorded. An iconic image of silicon dumbbells (Figure 4.9.2) is shown in a 2001 paper.¹¹ The same instrument at IBM was used to record sub-Angstrom resolution images.²

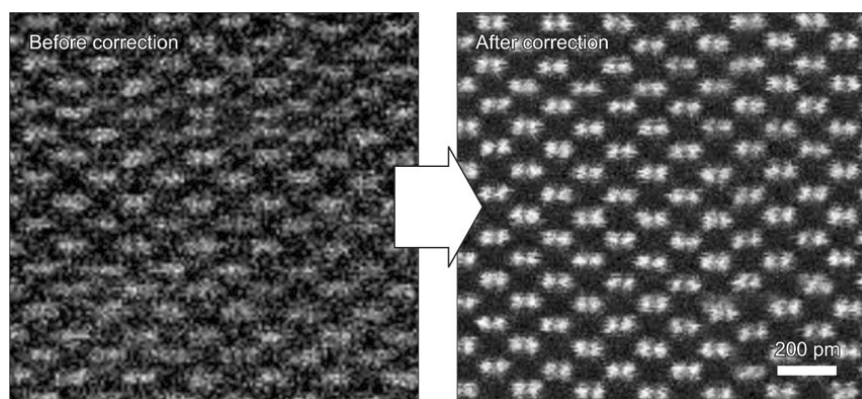


Fig. 4.9.2. Scanning TEM micrograph of silicon “dumbbells” before and after automatic aberration correction. *Source:* Inada H, *et al.* High-speed and sensitive x-ray analysis system with automated aberration correction scanning transmission electron microscope. *Applied Microscopy*. 2015;45(1):1–8. *Credit:* Creative Commons CC BY-NC 3.0.

1 As spherical aberration correction was being successfully imple-
2 mented in commercially available electron microscopes, chromatic
3 aberration was now the major hurdle to higher resolution. Many com-
4 mercial electron microscopy manufacturers simply limited the energy
5 spread of the incident beam by using a monochromator which mini-
6 mized but did not correct for chromatic aberration.

7 After a workshop convened by Murray Gibson in July 2000, the
8 TEAM (Transmission Electron Aberration-corrected Microscope)
9 project was funded by the US Department of Energy involving
10 the national laboratories at Argonne, Brookhaven, Oak Ridge, and
11 Lawrence Berkeley as well as the Frederick Seitz Materials Research
12 Laboratory at the University of Illinois. Harald Rose's new design
13 replaced each sextupole in the original corrector by a telescopic
14 quadrupole-octupole quintuplet. CEOS further optimized the design
15 creating an achromatic aplanatic system with stabilities at the order
16 of 2×10^{-8} for all electric and magnetic quadrupole fields. FEI and
17 CEOS built two instruments, TEAM 0.5, a TEM and STEM with
18 spherical aberration correction, and TEAM 1, a STEM with spheri-
19 cal aberration correction and TEM with chromatic and spherical
20 correction, which were installed at the National Center for Electron
21 Microscopy at the Lawrence Berkeley National Laboratory in 2008.
22 These best-in-class instruments have resolutions of 65 and 55 pm
23 at 200 and 300 kV,¹⁷ respectively, and the STEM resolves a 47 pm
24 dumbbell spacing along the 114 direction in a germanium crystal.¹³

25 Samples containing light elements such as carbon, oxygen, or
26 lithium degrade rapidly in an electron beam due to "knock-on"
27 damage, where atoms are displaced from their original positions
28 by high-speed electrons. However, reducing the accelerating volt-
29 age increases the damage due to radiolysis and chromatic aberr-
30 ation becomes dominant. At the University of Ulm in collaboration
31 with two companies, FEI and CEOS, the SALVE (Sub-Ångstrom
32 Low-Voltage Electron microscopy) project initiated by Ute Kaiser
33 has shown spherical and chromatic aberration correction for accel-
34 erating voltages between 20 and 80 kV.²² Between 40 and 80 kV
35 sub-Ångstrom resolution was achieved. More recently, a Japanese col-
36 laboration by the microscopy manufacturer JEOL and the National

Institute of Advanced Industrial Science and Technology built a TEM with a spatial resolution of 1.4 Å at 15 kV.²⁵ This allowed the observation of a monolayer of graphene at atomic resolution. This bodes well for the structural characterization of very beam-sensitive materials.

Over half a century of experimental struggles since the emergence of electron microscopes in 1932 allowed us to finally achieve the goal of sub-Ångstrom imaging. Although the key strategy, namely the deviation from round electro-optical elements by using multipoles, was recognized early on by Scherzer in 1947 and the first corrector was built by Robert Seeliger in the early 1950s, we had to wait until the 1990s until we could control the mechanical and electronic instabilities that were limiting the resolution. Only after these “secondary effects” were understood and controlled did aberration limit the resolution and could be corrected for. The length of such developments tests the patience of private and public funding agencies; however, transformative scientific advances are more and more becoming battles of attrition rather than singular strokes of geniuses.

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